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THE MECHANISM OF STRENGTH LOSSES IN NYLON 6.6 PARACHUTE MATERIA--ETC(U)

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MELBOURNE, VICTORIA

REPORT

MRL-R-801

THE MECHANISM OF STRENGTH LOSSES IN NYLON 6,6
PARACHUTE MATERIALS AFTER SALT WATER CONTAMINATION

G.T. Egglestone

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10) G.T./Egglestone

ABSTRACT

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Tensile strength losses in nylon 6,6 suspension lines contaminated with seawater result from abrasion damage due to salt crystals that penetrate the weave structure and cause shearing of the nylon filaments. This is contrary to a previous mechanism (2) that attributed these losses to the increased local relative humidity and moisture uptake caused by deliquescent salts, in particular magnesium chloride ($MgCl_2 \cdot 6H_2O$), present in seawater. Analysis using the Rheovibron Viscoelastometer has shown that the presence of $MgCl_2 \cdot 6H_2O$ on nylon 6,6 material does not increase the local relative humidity but decreases it, leaving the nylon in a drier state.
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16. ABSTRACT (If this is security classified, the announcement of this report will be similarly classified):

Tensile strength losses in nylon 6,6 suspension lines contaminated with seawater result from abrasion damage due to salt crystals that penetrate the weave structure and cause shearing of the nylon filaments. This is contrary to a previous mechanism [2] that attributed these losses to the increased local relative humidity and moisture uptake caused by deliquescent salts, in particular magnesium chloride ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$), present in seawater. Analysis using the Rheovibron Visco-elastometer has shown that the presence of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ on nylon 6,6 material does not increase the local relative humidity but decreases it, leaving the nylon in a drier state.

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THE MECHANISM OF STRENGTH LOSSES IN NYLON 6,6
PARACHUTE MATERIALS AFTER SALT WATER CONTAMINATION

1. INTRODUCTION

In the past, airdropping of service personnel into water was considered a specialist operation. Today in Australia it is an accepted part of the parachutists basic training. Although most training descents are into freshwater, sea parachuting is still common, particularly in the training of airservice crewmen.

Residual seawater contamination affects the mechanical properties of textile materials and for this reason any contaminated parachute assembly must be washed and dried in accordance with the Parachute Maintenance Manual [1]. If this washing procedure is inadequate, staining and localised stiffness of the parachute assembly results, accompanied by a reduction of up to 25% in the tensile strength of suspension lines.

An understanding of the mechanism of the observed strength losses is of importance in establishing a safe maintenance procedure for water descent parachutes. From an earlier investigation of the effect of seawater contamination on the mechanical properties of parachute cord and webbing [2] it was concluded that strength losses arose from the presence of deliquescent salts in the contaminated materials. The authors claimed that these salts produced an increase in relative humidity which resulted in a lower tensile strength of the nylon. An alternative process which leads to strength loss through the cutting action of residual salt crystals was considered not to occur. The favoured mechanism suggests that an adequate washing procedure would result in complete strength recovery. In contrast, a mechanism involving the cutting action of residual salt crystals would result in permanent strength losses after stressing or handling of the dry, contaminated line. Strength losses caused by this mechanism would be irrecoverable on washing. To determine the strength losses which occurred due to seawater contamination and to elucidate the mechanism of failure, the following investigations were undertaken:

- (i) measurements of strength losses for suspension lines after simulated seawater immersion
- (ii) measurements of strength losses for suspension lines in the presence of magnesium chloride ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$) and sodium chloride (NaCl)

- (iii) scanning electron microscopy of fracture surfaces of samples from (i) and (ii)
- (iv) detailed investigation of the effect of artificial seawater and deliquescent salts on the modulus of nylon 6,6 fibres using dynamic mechanical measurements.

2. EXPERIMENTAL

2.1 Materials

- (i) The braided nylon suspension line was manufactured to specification DTD 5620 cord number CB 203. It was constructed using high tenacity bright nylon yarn. The minimum specified breaking strength for the line was 1.8 KN.
- (ii) The nylon 6,6 taffeta material was a plain woven cloth of 44.5 g/m^2 , having 44 ends/cm and 39 picks/cm. The nylon yarn used in the construction of the cloth had a linear density of 5.3 tex and consisted of 13 filaments each having a diameter of 20 μm .
- (iii) The moisture content of the samples was determined in accordance with ASTM Part 32. Method D2654-76 [3].

2.2 Immersion Techniques to Simulate the Effects of Contaminants in Seawater

Suspension lines were immersed in the following solutions:

- (i) concentrated simulated seawater solution [4] (five times the recommended concentration was used to simulate the effect of concentration build-up on drying)
- (ii) saturated sodium chloride solution
- (iii) saturated magnesium chloride solution
- (iv) distilled water.

2.3 Mechanical Measurements

Tensile strength determinations (≥ 5 for each test) were done on suspension lines using the following two instruments:

- (i) Denison T41D tensile testing machine, at a constant rate of extension of 200 mm/min, fitted with 25 mm diameter bollard grips.

- (ii) Instron 1026 table model tensile testing machine, at a constant rate of extension of 200 mm/min, using horn grips. The Instron testing machine was also used for fatigue cycling to a constant load of 981N.

Suspension lines were tested after being subjected to the following pretreatments:

- (i) immersion in a solution of simulated seawater [4] for 16 hours, allowed to air dry, then conditioned at 65% relative humidity (RH) and 20°C for
 - (a) 24 hours
 - (b) one month
- (ii) immersion in saturated solutions of magnesium chloride or sodium chloride, allowed to air dry, then conditioned at room temperature for 16 hours
- (iii) immersion in distilled water and tested whilst wet.

2.4 Scanning Electron Microscopy

A fracture surface of original suspension line was compared to those resulting from immersion in the following solutions:

- (i) artificial seawater solution [4]
- (ii) saturated sodium chloride solution
- (iii) saturated magnesium chloride solution.

The sample contaminated with magnesium chloride was wet when broken due to the deliquescent nature of the salt.

The lines were air dried prior to breaking.

2.5 Dynamic Mechanical Measurements

All measurements were done using a Rheovibron DDV-11-C Dynamic Viscoelastometer operated at a constant frequency of 110 Hz. The high temperature environmental chamber was used, the sample grips being modified as described by Massa et al [5] in order to overcome the problem of grip slippage which occurs during tensioning of the sample. The temperature was monitored using a chromel/alumel thermocouple held near the sample surface.

Nylon 6,6 taffeta material was used in all dynamic mechanical measurements. Measurements of the complex modulus $|E^*|$ and $\tan \delta$ at a constant temperature of 20°C and various relative humidities (RH) were made. The samples were preconditioned to the required RH, these conditions being maintained in the apparatus by placing a pool of the relevant saturated salt solution [6] beneath the sample. Solid phosphorus pentoxide was used to obtain a RH of 0%.

The temperature dependant of $|E^*|$ and $\tan \delta$ was determined for two samples of nylon material, one unconditioned the other preconditioned to 100% RH. The unconditioned sample of nylon 6,6 material was tested from 0°C to 130°C using no humidity control and a heating rate of 2°C/min. The result obtained was considered to be typical of a dry nylon sample. The temperature dependance of nylon 6,6 at saturation was determined by preconditioning a sample to 100% RH and testing over a temperature range of -6°C to 24°C. Distilled water was placed beneath the sample to maintain 100% RH throughout the experiment. The sample was cooled to -6°C by circulating chilled methanol through the chamber, then heated to 24°C at 1°C/min.

3. RESULTS

3.1 Strength Losses after Simulated Seawater Immersion

After immersion in simulated seawater, the suspension line showed significant stiffening, and microscopical examination showed the presence of salt crystals within the outer sheath. Tensile strength values of the complete suspension line are summarized in Table 1. It can be seen that after 24 hours the strength loss was 28.3% and this did not alter significantly after standing for one month.

The moisture content of the samples after immersion increased from 3.0% to 4.2%, which is indicative of the deliquescent nature of the salts that had accumulated in the suspension line.

After rinsing the lines in distilled water, 94% of the original tensile strength was recovered. The moisture content of the line (refer Table 1) indicate that the deliquescent salts were removed and microscopic examination showed the line to be completely free from contamination by salt crystals.

3.2 Strength Losses after Immersion in NaCl and MgCl₂ Solutions

In an attempt to determine the reasons for the strength losses observed in Table 1, samples of suspension line were immersed in saturated solutions of MgCl₂ and NaCl. The breaking strengths and elongation to break of the outer sheath component were determined and are shown in Table 2. Also shown are the values obtained for samples immersed in artificial seawater and distilled water.

It can be seen that the breaking strength for the sample treated with MgCl₂ was reduced by only 6.6% from that of the original sample. This sample was wet on testing due to the absorption of water by the magnesium chloride crystals. The strength loss observed from a sample saturated with water was 12.9%. The reason for this observation will be discussed later. Clearly the observed strength loss of 26.5% from the sample treated with seawater cannot arise solely from the pressure of deliquescent salts such as MgCl₂·6H₂O.

The sample treated with NaCl showed a strength loss of 16.3% compared with the original sample. This loss was attributed to the abrasive nature of the cubic NaCl crystals that were observed between the filaments in the line structure. If the decrease in tensile strength results from shearing of the filaments by these crystals, the cycling of a NaCl contaminated suspension line to 50% of its ultimate tensile strength should result in a loss in strength consistent with cumulative abrasion damage. Results in Table 3 show that on progressive loading of the suspension line there is a decrease in the tensile strength for both the seawater and NaCl treated samples.

3.3 Scanning Electron Microscopy (SEM) Examination of Fibres

Examination of the fracture surfaces of the samples tested as shown in Tables 1 and 2 was performed using SEM. Figure 1(a) shows a typical failure surface of a fibre from the original suspension line. The ductile nature of the failure is clear. Figure 1(b) is typical of a NaCl contaminated suspension line. The cubic crystals of NaCl are evident, there is significant surface damage to the fibre, and the failure surface shows reduced plastic deformation. Closer examination of the failure surface (Figure 1(c)) shows the deposited crystals and evidence for the cutting action of these crystals on the filaments.

Filaments from a sample treated with MgCl₂ show none of these features and as can be seen from Figure 1(d) the failure mode is ductile. Figure 1(e) shows a typical fracture surface from a seawater immersed sample and it appears that the failure mode is similar to that in Figure 1(b).

3.4 Dynamic Mechanical Measurements

Examination of nylon 6,6 using dynamic mechanical techniques allows a study of the polymer in the solid state and detection of the temperature at which any relaxation processes occur. The Rheovibron DDV-11-C Dynamic Viscoelastometer was used to determine the complex modulus $|E^*|$, damping or loss factor, $(\tan \delta)$ and glass transition temperature, (T_g) , of the nylon 6,6 taffeta material.

The principle of operation of the Rheovibron Viscoelastometer is described in detail in a number of articles [5, 7, 8]. In general the operation consists of a sinusoidal strain being applied to one end of a sample causing a force to be generated at the opposite end. The magnitude of the stress and strain and the phase angle between them are then measured. From this, the complex modulus $|E^*|$ and the loss factor $\tan \delta$ can be calculated.

The two components of the complex modulus $|E^*|$ are the storage modulus E' and the loss modulus E'' . This relationship is represented by

$$|E^*| = E' + iE''$$

where

$$E' = |E^*| \cos \delta$$

$$E'' = |E^*| \sin \delta$$

Thus

$$\tan \delta = \frac{E''}{E'},$$

and

$$E' \approx |E^*| \quad (\text{as } \delta \text{ is generally small})$$

In the Rheovibron studies it was convenient to measure the absolute value of the complex modulus, $|E^*|$ and the phase angle δ as $\tan \delta$. Therefore for these experiments the glass transition temperature, T_g , was defined in terms of the maximum in $\tan \delta$.

3.5 Effect of Water on the Dynamic Mechanical Properties of Nylon

Nylon 6,6 being a semicrystalline polymer, consists of both crystalline and amorphous regions which have intermolecular hydrogen bonds between the polymer chains. Due to crystal packing, water is absorbed mainly in the amorphous region where it disrupts these hydrogen bonds [8-12] resulting in a decrease in the glass transition temperature, T_g , Young's Modulus, and the tensile strength (refer Table 2). Figure 2 shows the reduction in both T_g and $|E^*|$ with increased moisture content. The T_g for nylon 6,6 taffeta material was reduced from 122°C for a sample as received, to 9°C for a sample conditioned at 100% RH for 7 days.

The modulus value $|E^*|$ was similarly affected and changes from 6.5×10^9 Pa for a sample conditioned at 0% RH, to 1.3×10^9 Pa for a sample at 100% RH. The values for $\tan \delta$ versus RH at a fixed temperature of 20°C are given in Figure 4. A correlation coefficient of 0.988 was obtained for the linear portion between 0% and 60% RH.

3.6 Effect of the Deliquescent Salt $MgCl_2 \cdot 6H_2O$ on the Modulus $|E^*|$ and T_g

As previously mentioned, modulus values are very sensitive to change in moisture content. It was thus possible to determine the effect the deliquescent salt $MgCl_2 \cdot 6H_2O$ had on the nylon fibres. To demonstrate this effect a sample of original nylon 6,6 taffeta was sandwiched between layers of $MgCl_2 \cdot 6H_2O$ crystals and allowed to stand overnight. When tested at a constant temperature of 20°C the modulus $|E^*|$ was found to be 5.1×10^9 Pa. Figure 3 shows that a modulus value of this magnitude was typical of a sample of nylon taffeta equilibrated to 44% RH.

The above results show that the sample of nylon 6,6 taffeta material contaminated with $MgCl_2 \cdot 6H_2O$ crystals does not absorb water, but that the presence of the hydrated crystals creates an equilibrium condition consistent with that of a saturated $MgCl_2$ solution.

4. CONCLUSIONS

Tensile strength determinations on nylon 6,6 suspension lines contaminated with $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, NaCl and artificial seawater [4] gave strength losses of 6.6%, 16.3% and 26.5% respectively. The determined value for the sample contaminated with artificial seawater is similar to that found in practice, where strength losses of up to 25% have been reported. Results have indicated that the strength lost by the samples treated with artificial seawater cannot be attributed to the deliquescent nature of the $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ present. This is confirmed by examination of the fracture surfaces of the above samples using scanning electron microscopy which shows the mode of failure of samples contaminated with NaCl and artificial seawater to be similar. They show less plastic deformation when compared to the sample contaminated with $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ which has a fracture surface similar to that observed for undamaged nylon.

The mechanism of failure for the sample contaminated with $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ was determined using the Rheovibron at varying conditions of RH which confirmed that the modulus of nylon 6,6 decreases with increasing moisture content. The modulus value for the nylon sample contaminated with $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ increased showing the nylon to be in a drier state than it was originally. This is thought to be caused by the deliquescent $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ crystals on the sample surface which in the process of attaining chemical equilibrium remove water from the nylon.

Therefore the observed strength losses in the nylon suspension lines was attributed to the abrasive nature of the salt crystals present in seawater and not to any increase in moisture content due to the presence of deliquescent salts. Fatigue cycling of suspension line contaminated with both NaCl and artificial seawater confirmed this and showed strength losses consistent with cumulative abrasion damage.

5. ACKNOWLEDGEMENTS

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TABLE 1
TESTING OF COMPLETE CORD ASSEMBLY

Test	Original	Immersed Artificial Seawater 5x AS1580 Conditioned 24 hours	Washed 5x Distilled H ₂ O Conditioned 24 hours	After Conditioning One Month	
				Immersed Artificial Seawater 5x AS1580	Washed 5x Distilled H ₂ O
Average Breaking Load N x 10 ³	2.11	1.51	1.97	1.39	1.93
% Loss		28.4	6.3	33.8	8.2
Standard deviation δ	5.5	*23.2	8.3	*25.8	15.9
Moisture %	3.0	4.2	3.2	4.2	2.7
Microscopical Examination		crystals are present and have penetrated the outer sheath	fibres are completely free from contamin- ation		

Note: All samples were air dried then conditioned at 65% RH and 20°C

* The large standard deviation δ values for samples immersed in artificial seawater shows the scatter of breakages expected for samples contaminated with salt crystals.

TABLE 2

TESTING OF OUTER SHEATH

Solution used for immersion	Tensile Strength $N \times 10^3$	Extension %	Loss %
Original sample	1.82	33.6	-
Saturated NaCl	1.52	29.5	16.3
Saturated $MgCl_2$	1.71	29.4	6.6
Artificial seawater 5 x AS1580	1.34	28.3	26.5
Distilled H_2O (tested wet)	1.59	33.4	12.9

TABLE 3

TESTING OF SUSPENSION LINE AFTER PROGRESSIVE LOADING

Number of Progressive Loadings to 981N	Original Cord		Immersed 5X AS1580		Immersed Saturated NaCl	
	Breaking N x 10 ³	Loss %	Breaking N x 10 ³	Loss %	Breaking N x 10 ³	Loss %
ORIGINAL 0	1.96	0	1.84	0	1.84	0
1	1.89	3.5	1.78	3.2	1.76	4.2
3	1.91	2.5	1.50	18.6	-	-
5	1.86	5.0	1.42	22.9	1.72	6.9
10	1.81	7.5	1.43	19.7	1.50	18.6

* The samples were cycled to 50% of their ultimate breaking strength, washed to remove salt contamination, dried, conditioned in air, then broken.



a



b



c



d



e

FIG. 1 - Scanning electron micrographs of single fibres from nylon 6,6 suspension lines.

- (a) Fibre undamaged.
- (b) Fibre contaminated with NaCl.
- (c) Fibre contaminated with NaCl. NaCl crystals are evident.
- (d) Fibre contaminated with $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$.
- (e) Fibre contaminated with simulated seawater.

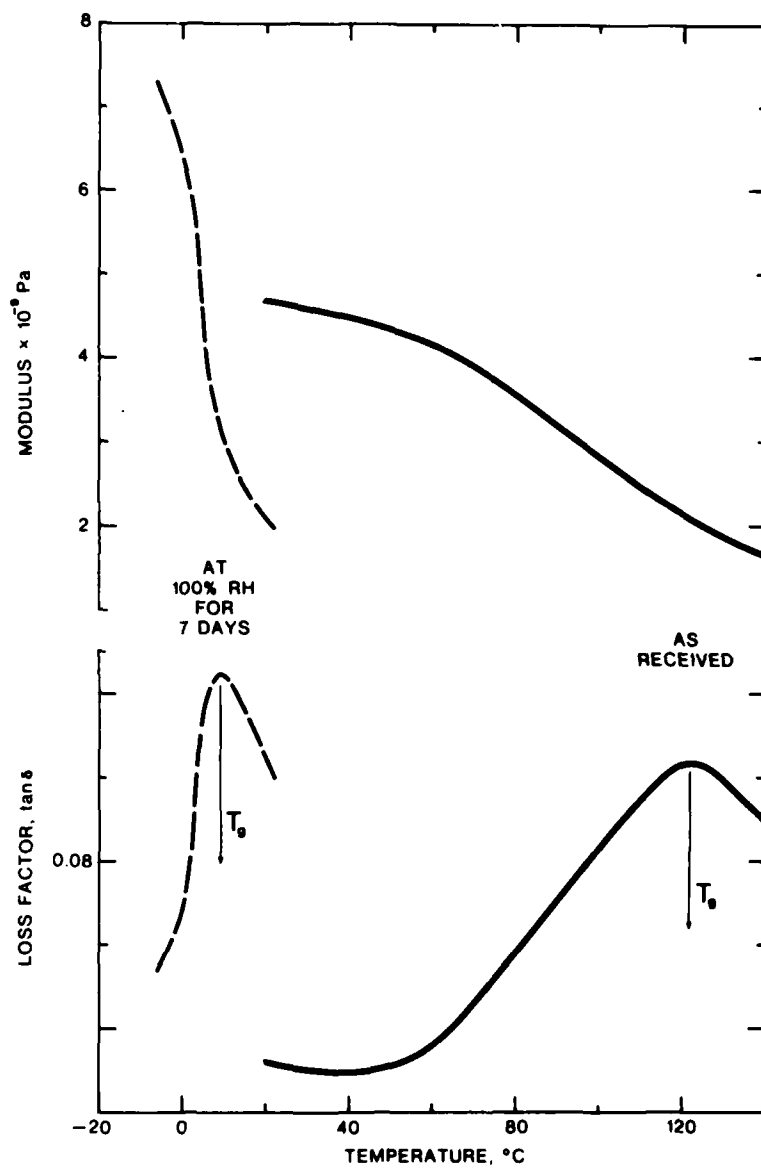


FIG. 2 - The effect of water on the modulus and T_g of nylon 6,6 taffeta material as a function of temperature.

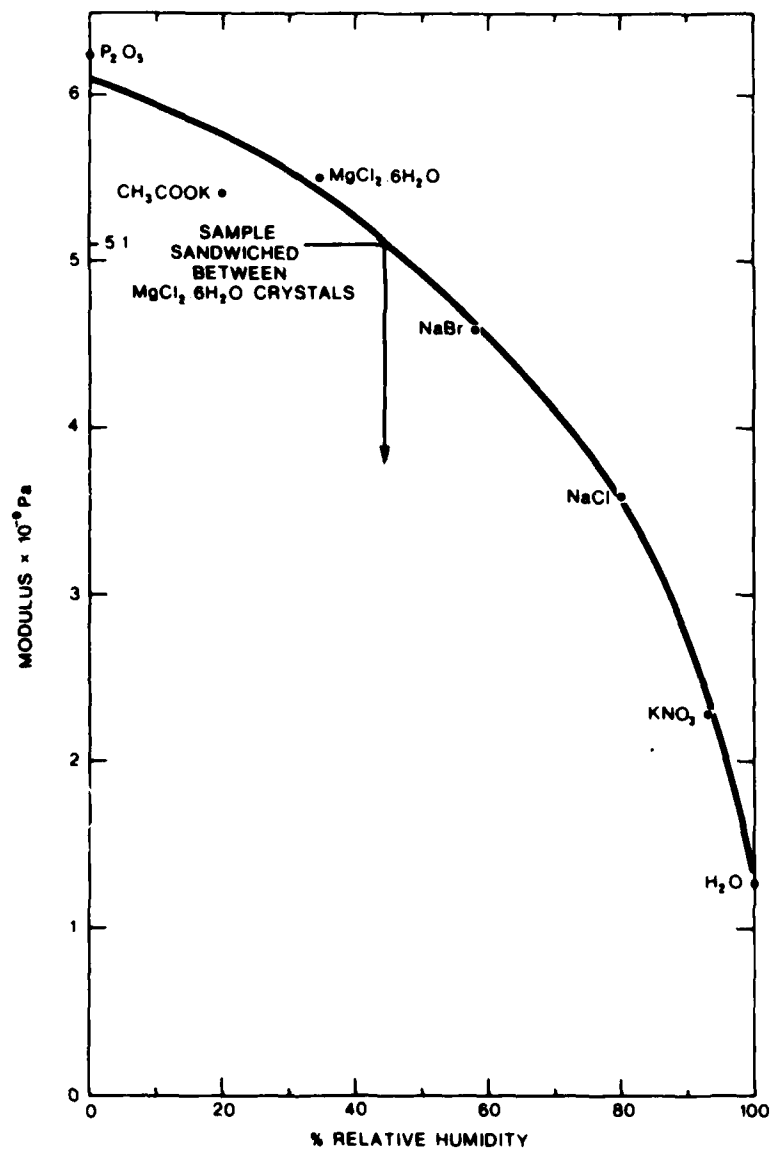


FIG. 3 - The modulus of nylon 6,6 taffeta material as a function of relative humidity.

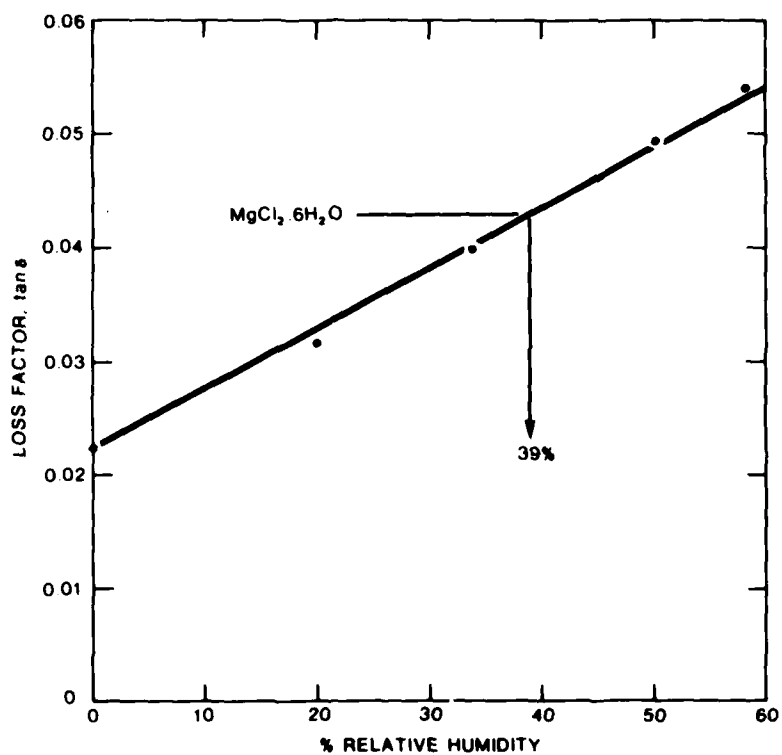


FIG. 4 - The change in $\tan \delta$ of nylon 6,6 taffeta material as a function of relative humidity.

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